California Environmental Protection Agency

Air Resources Board

Northern Laboratory Branch
Monitoring and Laboratory Division

SOP MLD SAS06

PROCEDURE FOR THE QUALITATIVE DETERMINATION OF COMPOUNDS IN CONSUMER PRODUCTS BY HEADSPACE GAS CHROMATOGRAPHY / MASS SPECTROMETRY

February 16, 2005, Revision 1.2

DISCLAIMER: Mention of any trade name or commercial product in Method 310 and associated Standard Operating Procedures does not constitute endorsement or recommendation of this product by the Air Resources Board. Specific brand names and instrument descriptions listed in the Standard Operating Procedures are equipment used by the ARB laboratory. Any functionally equivalent instrumentation can be used.

1 INTRODUCTION

The analysis of volatile organic compounds (VOCs) in consumer products by SAS07 provides a quantitative analysis of the compounds present in a particular consumer product. To characterize the volatile content present, this procedure qualitatively identifies compounds present in a consumer product.

2 SUMMARY OF METHOD

Approximately 100 µl of undiluted sample is introduced into a 40 ml headspace vial containing 4.0 ml of 10% water/polyethylene glycol 400 (PEG 400). The vial is then sealed and placed into the headspace sampling unit. The PEG 400 dispersant is used to insure that small variations in sample polarity will not affect the analyte(s) partition coefficient. The vial is equilibrated to 80 ^{o}C, pressurized to a constant pressure, and an aliquot of the headspace gas is injected into a Gas Chromatograph/Mass Spectrometer (GC/MS).

3 INTERFERENCES AND LIMITATIONS

The headspace vial must be checked after sealing to ensure leakage does not occur. The vial should never be heated to a temperature exceeding the boiling point of the dispersant (approximately 100 ^{o}C) as this may cause a rapid increase in pressure within the vial.

Cross contamination can occur whenever high-level and low–level samples are analyzed sequentially. Whenever a high-level sample is analyzed, it must be followed by a blank to ensure that contamination has not occurred.

4 INSTRUMENTATION AND EQUIPMENT

4.1 Gas Chromatographic System

4.1.1. Headspace Sampling System equivalent to Hewlett-Packard Model 7694.

Vial Oven Temperature:	80 ^{o}C
Loop Temperature:	120 ^{o}C
Transfer Line Temperature:	180 ^{o}C
Loop Size:	1 ml
Transfer Gas Flow:	>30 ml/min
Vial Pressure:	15 psi;

4.1.2 GC/MS System equivalent to Hewlett-Packard Models 5890/5971.

GC/MS Parameters are as follows:

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Mass Range:	40-300 amu
Initial Column Temperature:	40 ^{o}C
Initial Hold Time:	8 min.
Column Program Rate:	10 ^{o}C/min
Final Column Temperature:	250 ^{o}C
Injector Temperature:	250 ^{o}C
MS Interface Temperature:	280 ^{o}C
Helium Carrier Gas Pressure:	17 psi @ 50 ^{o}C (linear velocity: 26 cm/sec)
Spilt Ratio:	40/1 (split flow > than transfer flow);

4.2 GC Column: DB-VRX, 60m x 0.25 mm i.d. with 1.4 μm film;

4.3 Headspace vials, 40 ml with crimp-top caps;

4.4 Crimper for (4.3);

4.5 Rainin pipettors, 250 μl and 10.0 ml with pipette tips.

5 REAGENTS AND MATERIALS

5.1 Dispersant – 10% water/polyethylene glycol 400 (PEG 400) – prepared by adding 50 ml reagent grade water to 450 ml PEG 400 and mixing well.

5.2 Gases – ultrahigh purity helium (He), and compressed air.

6 PROCEDURE

6.1 Prepare a Blank by placing 4.0 ml PEG 400 into a 40 ml vial and cap the vial.

6.2 Prepare samples by placing 100 μl of undiluted sample into a 40 ml vial containing 4.0 ml PEG 400 and cap the vial.

6.3 Place the vials in the auto sampler for the headspace unit and start auto sampler unit.

6.4 Set up GC/MS acquisition software and start run.

7 QUALITY CONTROL

A PEG 400 blank should be analyzed to note any contamination that may be present in the system.

APPENDIX A

Headspace Screen Analysis

The headspace analysis is run on a Gas Chromatograph/Mass Spectrometer (GC/MS) system equivalent to Hewlett Packard Models 5890/5971 equipped with a 60 m x 0.25 mm id, 1.4 μm film DB-VRX capillary column.

Check the He tank pressure to ensure there is enough for the analysis.

1 Sample Preparation

1.1 Label clean 40 ml vials with the appropriate sample number(s). Assemble crimp caps by placing Teflon lined septum into the caps with the Teflon side down.

1.2 Pipette 4.0 ml of dispersant (10% H_2O/PEG 400 solution) into each vial.

1.3 Pipette 100 μl of undiluted sample into the dispersant. Cap the vials.

1.4 Load the prepared vials in numerical sequence into the headspace auto sampler tray. The tray advance button can be used to advance the vial holders so that the #1 position is accessible.

1.5 On the auto sampler keypad, select “vial parameters.” The display will show “first vial =1.”

1.6 Press ↑ or ↓ buttons to scroll through the settings on the display until “last vial = #” appears.

1.7 Enter the number corresponding to the total number of vials to be run and then press “enter.”

1.8 To start the auto sampler, press the start/stop button.

2 GC/MS Software Preparation

2.1 On the PC, select GC/MS instrument #1 MSTop/enhanced – HDSP2.M/HDSP.S window.

2.2 Load Method.

2.2.1 Under **Method**, select ***Load Method.***

2.2.2 Click on ***HDSP2.m***, then ***OK***.

2.3 Load Sequence.

2.3.1 Under ***Sequence***, select ***Load Sequence.***

2.3.2 Click on ***HDSP.s***, then ***OK***.

2.4 Modifying Sequence.

2.4.1 Under ***Sequence***, select ***Edit Sample Log Table***.

2.4.2 Click on a line to edit it.

2.4.3 Line 1 - At the keyword string, enter month (alpha) and day (numeric) of the run followed by “01”. Example Feb1601.

2.4.4 Line 2 to the end of your samples.

2.4.4.1 Under ***Type*** enter ***Sample.***

2.4.4.2 Under ***Vial*** the number is incremented.

2.4.4.3 ***Data File,*** leave blank.

2.4.4.4 Under ***Method*** type HDSP2.

2.4.4.5 Under ***Sample Name*** enter lab sample ID.

2.4.4.6 ***Miscellaneous Information*** can be left blank.

2.4.5 The first sample should be a PEG 400 blank.

2.4.6 Click on ***Cut*** to delete a highlighted line from the Sample Log Table.

2.4.7 To add more lines to the Sample Log Table, highlight the last line and click on the ***Repeat*** button as many times as necessary.

2.4.8 Click on ***OK*** when done.

2.5 Save the sequence.

2.5.1 Under ***Sequence***, select ***Save***, then ***OK***.

2.5.2 Select ***OK*** for the “Overwrite” question.

2.6 Print the sequence.

2.6.1 Under ***Sequence***, select ***Print Sequence***.

2.6.2 Select ***Brief Format***; then ***OK***.

2.7 Run the sequence.

2.7.1 Under ***Sequence***, select ***Run***.

2.7.2 Method Sections to Run, select ***Full Method***.

2.7.3 On a Barcode Mismatch select ***Inject Anyway***.

2.7.4 Overwrite Existing Data Files, check box if you want existing files with same file names as your samples to be overwritten.

2.7.5 Sequence Comment, Leave blank.

2.7.6 Operator Name, enter your initials.

2.7.7 Data File Directory, C:\HPCHEM\1\DATA\2005 or current year.

2.7.8 Click on ***Run Sequence.***

2.8 Each sample runs for approximately 30 minutes.

3 Data Analysis

3.1 Open the “Enhanced Data Analysis” window.

3.2 Loading Data File.

3.2.1 Under ***File***, select ***Load Data File***.

3.2.2 Use the scroll bar in the table on left-hand side of the screen to find your file of interest.

3.2.3 Click on the ***file name***, then ***OK***. The chromatogram of that sample will appear.

3.3 Print Chromatogram.

3.3.1 Under ***File***, select ***Print***.

3.3.2 Select ***Selected Window***; then ***OK***. The chromatogram is window "2."

3.4 Observe spectrum of a peak.

3.4.1 In the chromatogram window, double right click on peak of interest. The spectrum corresponding to the peak of interest will appear in the bottom window.

3.5 Determine possible spectrum matches from NIST library.

3.5.1 Double right click in the spectrum window. A library search of the peak of interest will appear below the sample spectrum.

3.5.2 Compare the library spectrum with the spectrum of the peak of interest. "Qual" indicates the degree of match, with 100 being perfect.

3.5.3 Select ***Print*** to print spectrum.

3.5.4 Select ***Done*** to exit.

3.6 After reviewing and printing data, minimize "enhanced data analysis" window, do not close it.

3.7 Remove vials from auto sampler unit.

SOP Revision History

DATE	VERSION	NOTES
March 10, 1998	1.1	1 Adjusted document font to Times New Roman 12. Inserted appendix A formerly a stand-alone document.
February 16, 2005	1.2	Adjusted document font to Arial 12. SOP updated to reflect current practices. Corrected revision enumeration.